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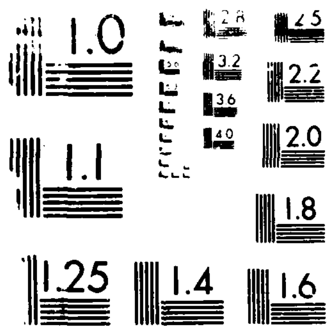
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TECHNICAL REPORT ARCCB-TR-87029

EVALUATION OF FILAMENT-REINFORCED ELECTROCOMPOSITES (REINFORCEMENT OF ELECTROFORMS WITH CONTINUOUS FILAMENTS)

AD-A188 649

V.P. GRECO

OCTOBER 1987

US ARMY ARMAMENT RESEARCH, DEVELOPMENT
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From a review of the literature, an updated evaluation of filament-reinforced electrocomposites is given and the benefits and limitations of the process are summarized. The successful results and properties obtained with electrocomposites from earlier studies are also presented and problems encountered are discussed. Process changes for improving the quality and strength of electrocomposites are proposed.

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INTRODUCTION

A critical problem in producing metal matrix composites (MMC) is the handling and damaging of brittle fibers due to high process temperatures and pressures. A process which does not require high temperatures or pressures is electrodeposition in aqueous solutions which offers a number of other benefits. Recognition of these benefits in the late 1960's resulted in numerous studies (refs 1-13) on the formation of filament-reinforced metal matrix composites by electrodeposition (i.e., electrocomposites). Since that time, much of the attention has been given to carbon filament-reinforced composites and to protective coatings on carbon, boron, and silicon carbide for high temperature environments.

The concept of fibrous-reinforced metal matrix composites may be regarded as one of the greatest material contributions of all times. Before the development of MMC, fibrous ceramics were usually considered unsuitable as candidate structural materials owing to their brittleness. Now the user can encapsulate these high strength and high modulus filaments into a suitable protective matrix to tailor the material to his specific needs. However, the success of high performance composites will depend not only on the quality of the filament, but on the process employed to fabricate the composite.

In previous studies on electrodeposition, most investigators were deeply concerned about composite properties, but gave little attention to electroforming techniques. The technique or plating practice employed plays a major role in the soundness and quality of the matrix metal encapsulating fibers or filaments. Therefore, a complete understanding of the electrodeposition of metals should prove invaluable for the fabrication of high performance composites.

References are listed at the end of this report.

OBJECTIVE

The purpose of this report is to provide an updated evaluation of filament-reinforced electrocomposites. In order to accomplish this, the important benefits and limitations of the process will be summarized, followed by a review of the literature on electroforming as a viable process to produce composites.

BENEFITS OF ELECTROCOMPOSITES

- No high temperatures or pressures are required in processing.
- A variety of composite forms can be produced with little or no post-machining required.
- Deposition rate and purity of the matrix metal are easily controlled.
- Filament spacing, tension, and volume are flexible and easily controlled.
- The filaments are easily pretreated (cleaned, etched, precoated, etc.) for maximum wetting and control of filament-matrix bonding.
- Finished composites have no size restrictions.
- The process can be interrupted and resumed at any time.
- Matrix properties can be changed by the use of pulse plating, periodic reverse, ultrasonics, bath additives, etc.
- Standardized interfaces (free of temperature or pressure effects) are provided for post-heating reaction studies.
- Several metals and alloys are available as candidates to be plated as matrix materials.

DISADVANTAGES OF ELECTROCOMPOSITES

- Not all matrix alloys or metals can be plated.
- Thick composite sections > 25 mm (1 inch) may be impractical for plating.

• Cross-wrapping of large diameter (> 4 mils) conductive filaments may require interim machining to eliminate voids.

• Complex forms may be impractical to produce.

From the above benefits, the absence of high temperature and pressure are the most important. Other major benefits are the ease of pretreating, spacing of filaments, and freedom of post-machining.

REVIEW OF PREVIOUS STUDIES

Discussion of problems encountered and opposing views in the literature, in reference to the above benefits, are as follows.

Electrodeposition of Matrix Alloys

In their assessment of electroforming, Schoutens and Tempo (ref 14) indicated the inability to deposit alloys. On the contrary, several alloys are commonly applied through electrodeposition, some of which are commerically plated on a large scale such as brass, bronze, lead-tin, and nickel, cobalt, iron alloys, etc. However, some alloys are still in the experimental stage and many of the complex alloys cannot be plated at all.

The fundamentals of alloy deposition and some of their properties have been presented by several authors, such as Brenner (ref 15), Raub and Muller (ref 16), and Faust (ref 17). The principles and control variables which apply to alloy deposition, however, are more complex than single-metal deposition.

Impurities in Electrodeposits

Schoutens and Tempo (ref 14) also claimed the impurities in a metal matrix as a limitation of plating. From a strict sense, no material is impurity free. Safranek (ref 18) provides comprehensive data on impurities and effects in electrodeposits. However, properly plated metals can be more pure than thermally-prepared metals. In fact, some high purity metals listed in the Metals Handbook (ref 19) are electrolytic.

Electrodeposition of Oxidation-Resistant Alloys

Baker et al (ref 7) reported the inability to plate oxidation-resistant alloys. Although not all oxidation-resistant alloys are amenable to plating, it is also doubtful whether many can be thermally prepared as matrix materials to produce sound composites. A number of these alloys surveyed by Brenner (ref 15) may prove to be very successful as oxidation-resistant matrix materials, some of which may still be in the experimental stage. Chromium-iron (ref 20) and iron-nickel-chromium (ref 21) alloys have also been reported. In an earlier study, Browning et al (ref 22) electroformed cobalt-tungsten (35 percent) alloys with higher strength than HS-25 alloys above 1100°F which may be useful with modifications. In the same study, promising quaternary alloys of cobalt-tungsten-nickel-iron were also produced.

Chromium metal, which has high oxidation resistance, can also be plated as a protective coating onto filaments and/or onto the surface of composites. For more severe environments, chromium coatings on composites may be laser-treated to produce surface alloying with the matrix (refs 23,24).

BOND STRENGTH OF THE FILAMENT-MATRIX INTERFACE

A high order of adhesion on surfaces of refractory, ceramic, and noncrystalline materials is difficult to achieve compared to iron group metals, regardless of the process employed to encapsulate the filaments. In some cases it will be necessary to place considerable dependence on mechanical locking between matrix and filament by chemical etching and post-heat treatment. Reports of problems and solutions which confirm the above are as follows.

Tungsten Filaments

Cooper (ref 6) reported poor bond strength with the plating of copper on 100 μM (4 mil) diameter tungsten filaments. High adhesion on most refractory metal surfaces is very difficult because of their tenacious oxide-filmed surfaces. Heat treatment was suggested as a means to improve bond strength, but etching the filament was not mentioned.

Another factor which must be considered is the diameter of the filament. Larger diameter filaments require higher bond strength (refs 12,25) for optimum stress transfer:

$$S_B = \frac{\sigma_f}{2\ell/r} \qquad (1)$$

where S_B is the minimum bond strength required and σ_f is the filament strength.

Ahmad et al (ref 5) reported high strength values of various volume fractions of 12.5 μM (½ mil) diameter tungsten filaments in electrodeposited nickel without any cleaning or pretreatment of filaments (Figure 1). Withers and Abrams (ref 10) showed lower strengths with 2 mil tungsten filaments in nickel (Table I).

Boron and Silicon Carbide Filaments

Chuang (ref 26) reported a problem with delamination and poor bond strength of silicon carbide-nickel composites. Delamination and poor adhesion could be caused by high residual tensile stresses in the deposit, which could be due to organic bath impurities. However, as previously mentioned, high adhesion of metals on silicon carbide and boron filaments (which are semiconductors) is difficult to achieve. Therefore, chemical etching and heat treatment is highly recommended to improve bonding.

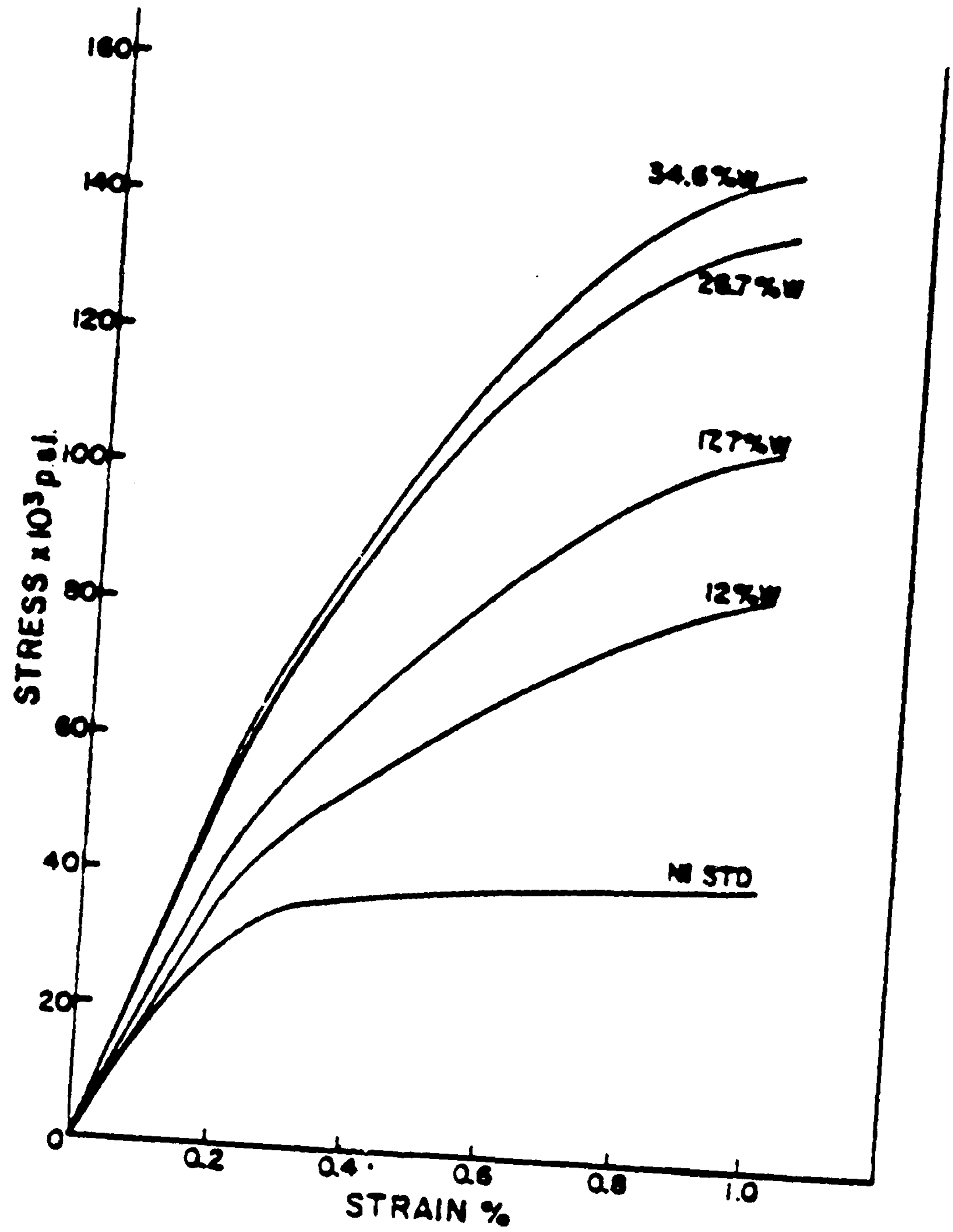


Figure 1. Strength of 12.5 μM (½ mil) tungsten-nickel composites (annealed).

TABLE I. MECHANICAL PROPERTIES OF VARIOUS ELECTROCOMPOSITE SYSTEMS

Composite System(ref)		V/O Fil	Filament Tensile Strength		Filament Modulus		Matrix Ultimate Strength		Matrix Modulus		Composite Tensile Strength		Composite Modulus		% Rule of Mixtures Strength Predicted	% Rule of Mixtures Modulus Predicted
			kg/mm²	10^3psi	kg/mm²	10^6psi	kg/mm²	10^3psi	kg/mm²	10^6psi	kg/mm²	10^3psi	kg/mm²	10^6psi		
4 mils B/Ni(9)		42	285	405	39790	56.6	42	61	16942	24.1	132	188	22918	32.6	42	87
		51	273	388	40212	57.2	63	90	17716	25.2	132	188	23621	33.6	80	85
4 mils B/Al(11)		15	210	300	38665	55	10	14.3	5273	7.5	25.4	36.2	9842	14	77	95.6
4 mils B/Al(10)		32	-	-			18	25.4*		7.8*	85	121	17575	25	-	-
4 mils B/Ni(10)	as plated	25	-	-	-	-	70	100	-	-	88	125	15466	22	-	-
	500°C	25		-	-	-	45	64	-	-	42	60	19684	28	-	-
5.6 mils B/Cu(26)	as plated	48	-	-	-	-	-	-	-	-	110	156	-	-	-	-
	heated 500°C	48	-	-	-	-	-	-	-	-	81	115	-	-	-	-
5.6 mils BN/Ni(26)	as plated	34	-	-	-	-	-	-	-	-	137	195	-	-	-	-
	500°C	34	-	-	-	-	-	-	-	-	85	1.	-	-	-	-
4 mils SiC/Ni(10)		34	-	-	-	-	70	100	-	-	107	152	30932	44	-	-
2-4 mils W/Ni(10)		56	352	500	35150	50	70	100			155	221	2956	42.1	68	
½ mil W/Ni(5)	as plated	34.3	352	500	35150	50	73	104	15466	22	149	212	21512	30.6	88.3	97
	650°C ½ hour	34.6	-	-	-	-	39	55	13638	19.4	117	166	21231	30.2		
C/Al(31)		-	-	-	-		8	11	-	-	23	32.4	-	-	-	-

A study on bond strength measurements by filament pull-out of boron and silicon carbide in electrodeposited nickel was reported by Greco et al (ref 12). Results have shown that the combination of filament etching and post-heat treatment significantly increased bond strength up to 500°C. Also, the tensile strength of single filaments of both boron and silicon carbide significantly increased with etching. In a later study (ref 13), the bond strength of nickel to nitride-coated boron filaments was measured, showing greater increases than boron (Table II). Wittich (ref 27) also found that nitride-coated boron filaments in nickel gave good results (Table I).

TABLE II. BOND STRENGTH OF NITRIDE-COATED BORON-NICKEL COMPOSITES

Filament	Heat Treatment	Average Bond Strength	
		Psi	kg/mm^2
Boron (BN)	None	2820	1.98
Boron	None	1830	1.29
Boron (BN)	500°C (970°F)	5310	3.73
Boron	500°C (970°F)	3206	2.25

Alexander and Stuhrke (ref 9) also reported increased bond strength with etching of boron filaments and heat treatment of boron-nickel composites.

Adsit (ref 8) attributed poor adhesion of boron-nickel composites to very close spacing of the filaments. The poor adhesion which may occur in this case is the joining of nickel to nickel from two adjoining filaments due to lateral growth morphology forming triangular voids between filaments (Figure 2).

Due to growth morphology on conductive filaments, specific steps must be taken to eliminate voids. A unidirectional row of filaments is first wound on a mandrel with careful spacing followed by complete encapsulation. Then a second layer of filaments is wound and spaced in the valleys formed between the filaments of the first row and encapsulated (Figure 3), etc. If nodules or any other irregularities from crystal growth form in the valleys, the following filament will unavoidably be misplaced, leading to possible void formation (refs 8,13).

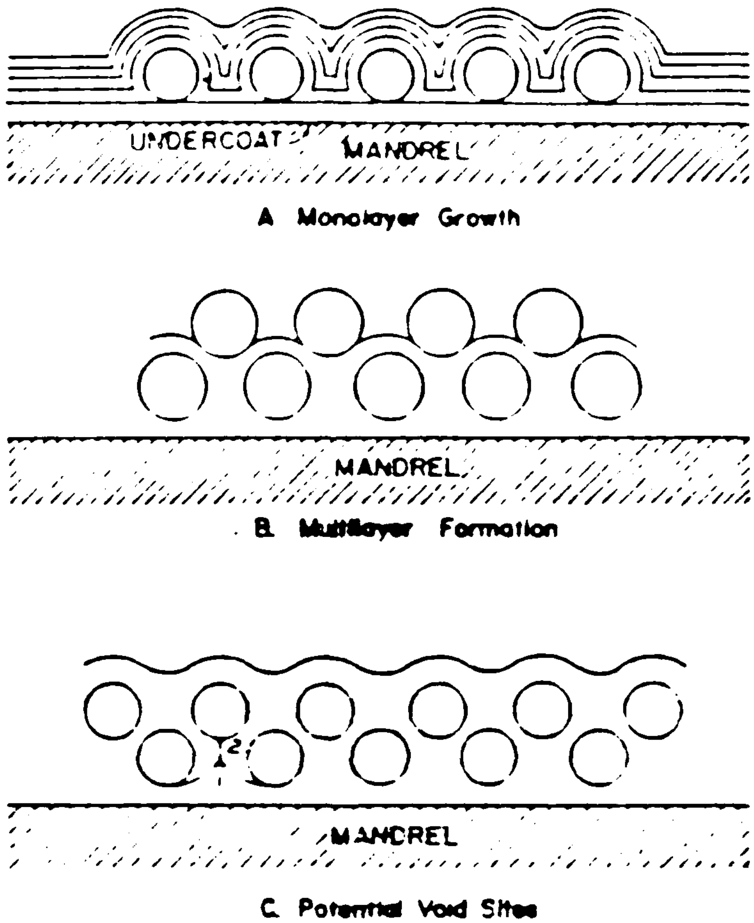


Figure 3. Electroformed composite of 100 μM (4 mil) filament layers (ref 9).

Sound electrocomposites with 42 volume percent (v/o) boron filaments were successfully produced. Composites with 56 v/o boron filaments contained 10 percent voids (ref 9).

A method (ref 13) used to eliminate voids with large diameter filaments was to remove the mandrel after each row of filaments was encapsulated, followed by removal of matrix surface irregularities by machining (Figure 4). The layered interfaces of the matrix are due to interrupted plating, however, the surfaces have been etched to improve the bond strength between layers.

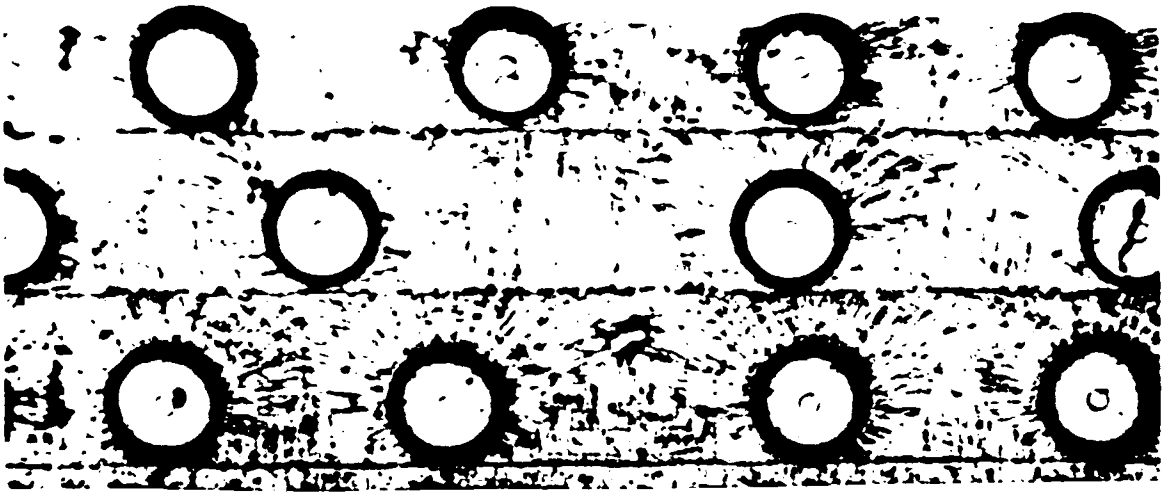


Figure 4. Boron-nickel composite with interrupted plating and machining of layers (100X).

With improvements in plating to eliminate voids, it is estimated that a minimum of one-filament diameter is necessary to produce a sound and continuous growth of deposit for complete encapsulation of the filament. Later studies (ref 13) showed complete encapsulation of 100 μM (4 mils) diameter nitride-coated boron nonconductive filaments with spacings as close as 25 μM (1 mil) (Figure 5). Harris et al (ref 28) encountered porosity problems with nonconductive filaments, but the diameter was 40 μM (1.6 mil) which obviously created a different growth pattern.

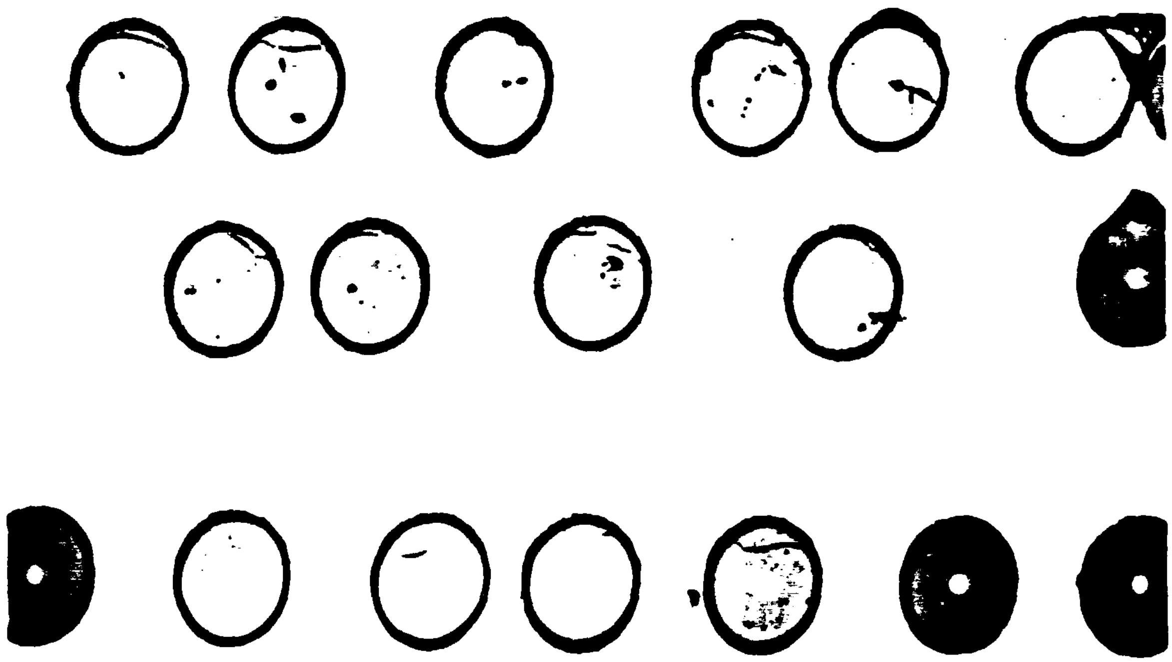


Figure 5. Nitride-coated boron-nickel electrocomposite with various filament spacing (100X).

Suchentrunk and Gammel (ref 29) encountered problems with the fabrication of a boron fiber-reinforced aluminum pressure vessel. It was designed as a hybrid construction (boron-aluminum cylinder with titanium shells) because of the stiffness problem with filament winding of boron. Suchentrunk (ref 30) also encapsulated boron carbide-coated boron fibers in copper, nickel, and aluminum.

Spacing of 50 μM (2 Mils) Conductive and Nonconductive Filaments

Studies (ref 6) with 50 μM (2 mils) diameter nonconductive filaments were shown to require a spacing of approximately 20 μM.

Baker et al (ref 7) produced electrocomposites with concentrations of 25-55 v/o 50 μM (2 mils) diameter tungsten filament in nickel and encountered increasing porosity with increasing filaments. The use of wetting agents, ultrasonics, and periodic reverse were suggested as possible measures to reduce porosity.

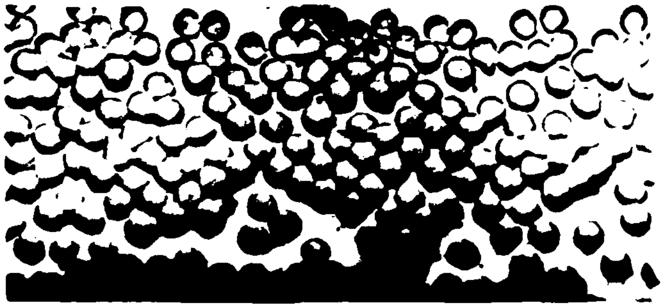
Spacing of 12.5 μM (½ Mil) Conductive Filament

Electrocomposites with 44 v/o 12.5 μM (½ mil) diameter tungsten filaments in a nickel matrix were successfully produced (ref 5) with just a few percent voids by simultaneous winding of filament during plating (Figure 6). The view after annealing shows that the composite is virtually free of voids.

During these studies it was found that the maximum diameter of conductive filaments which feasibly could be wound continuously during plating with minimal void formation was approximately 50 μM (2 mils). Cooper (ref 6) reported electrocomposites of 55 v/o 100 μM (4 mils) diameter of tungsten filaments in nickel, but failed to provide any data on voids or strength.

Conductive 12.5 μM (½ mil) tungsten filament has been shown to be an ideal reinforcement for encapsulation with minimal voids by electroforming. However, the high density of tungsten makes it a poor candidate where high strength-to-density ratios are required. The only available small diameter filament with a high modulus-to-density ratio is graphite filament.

As Plated



After Annealing

Figure 6. 44 v/o 12.5 μM (½ mil) tungsten filaments in nickel (250X).

CARBON AND GRAPHITE FILAMENT-REINFORCED ELECTROCOMPOSITES

As previously stated, composite studies in recent years have primarily concentrated on carbon or graphite filaments due to their high strength and high modulus properties at high temperature. Chemical reaction and structural recrystallization studies on graphite fibers coated with various metals showed deleterious effects in most cases (ref 14). Therefore, a number of studies were undertaken to further investigate the graphite/metal system. The majority of these studies have dealt with the plating of the individual fibers with a protective coating prior to fabrication of the composite by other processes dealing with high pressures and temperatures. Some of the interesting studies are as follows:

1. Meiser and Davison (ref 31) have achieved stability of fibers in an iron/aluminum alloy with a low solubility for graphite. They pointed out that without a high-strength, high-modulus graphite monofilament, difficulties in fabrication preclude the successful application of graphite in metal matrices.

2. Adsit (ref 8) reported poor copper/nickel composite strength results and attributed them to the low strength of the carbon yarn available at the time. He also stated that the nickel deposit did not fill all the void space in the yarn, and did not mention the pretreatment of the yarn, or how it was wound on the cathode mandrel.

Yarns consist of many fine monofilaments which are twisted together and, in most cases, are coated with some kind of organic sizing, since most yarns are made for a plastic matrix. Therefore, yarns with an unknown history should first be immersed in some suitable solvent to remove the sizing. Schmidt et al (ref 32) subjected graphite fibers to different solvents prior to plating.

A technique must be employed to unravel the yarn so that individual filaments are separated on the cathode mandrel for complete encapsulation. This procedure, although not a simple one, must be taken with all yarns or strands for producing good composites.

3. Shiota and Watanabe (ref 33) reported the nickel plating of polyacrylonitrile (PAN)-based carbon yarn followed by hot pressing of the plated filaments. The yarn consisted of 2000 monofilaments of 8 μM diameter, and they

claimed uniform encapsulation of monofilaments producing composites of high strength. The procedure that was employed to successfully encapsulate the individual filaments is reported in the Japanese literature (refs 34,35).

4. Some electrocomposites (refs 5,10) were produced with 10 μM diameter filaments of glass yarn in nickel, since they were available and their handling was similar to graphite (Figure 7).

When available, monofilaments should preclude the use of yarns or strands for ease of handling.

Prewound Filament　　　　Continuous Wound

Figure 7. Glass filament in nickel (100X).

5. Beetz and Schmidt (ref 36) reported the successful continuous nickel deposition onto carbon fibers for electromagnetic interference (EMI) shielding. The results show slight losses in tensile and modulus properties, but good compressive and short beam shear properties.

6. Jackson and Marjoram (ref 37) studied the compatibility of single carbon fibers coated with nickel or cobalt, and showed results to be applicable to bulk composites. Carbon fibers undergo structural recrystallization in contact with a nickel or cobalt matrix by a dissolution/diffusion/reprecipitation mechanism.

Graphitized fibers resist recrystallization better than carbonized fibers because their more orderly, stable structure is more resistant to dissolution.

7. Barlow and Sachs (ref 38) encapsulated 10 v/o carbon fibers in a lead-tin (93-7) matrix followed by hot pressing at 220 degrees and 10 ton/in.^{2} pressure. The shear strength of the composite was reported as 45.7 N/mm^2 compared to 26 N/mm^2 for the lead-tin matrix.

8. Chen et al (ref 39) plated carbon fibers with bi-metals of copper and nickel prior to encapsulation in aluminum by hot pressing for high temperature studies because of the low solid solubility of carbon in copper. Prior to plating, the fibers were desized by heating in nitrogen at 970°K followed by rinsing, immersing in solvents, and rinsing again. The coated carbon-aluminum composites with copper underlay were inferior to nickel or cobalt coatings alone at temperatures above 1020°K.

9. Baker et al (ref 40) reported on two methods of producing electrocomposites with carbon fibers; the fabrication of filament-wound components and the forming of warp sheets which can be bonded to form laminates. The latter process was quite successful.

10. Lewis and Walter (ref 41) reported on the electroforming of carbon or boron fibers in a nickel-cobalt matrix alloy by electroless plating. This is interesting since not many papers have been published on plating an alloy matrix.

11. Lavrenko et al (ref 42) plated copper on graphite and silicon carbide-coated graphite filaments.

MECHANICAL PROPERTIES OF ELECTROCOMPOSITES

Flat tensile specimens with continuous filaments can be conveniently prepared on oval mandrels (Figure 8). The cutting and trimming of composite test specimens are more difficult than conventional ones, since care must be taken to avoid filament damage.

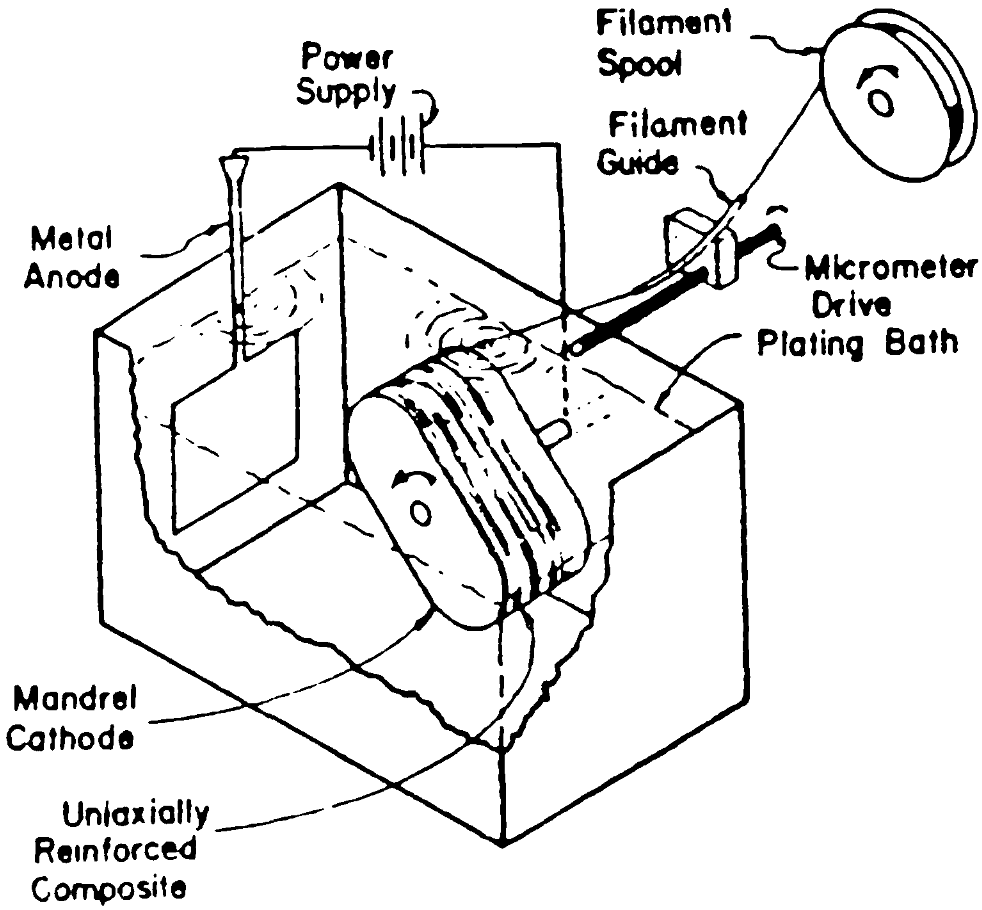


Figure 8. Mandrel design for producing flat tensile composite specimens (ref 10).

Strength measurements of continuous filament-reinforced electrocomposites with minimal voids compare favorably well with calculated values from the rule of mixtures. Table I shows some of the results of different electrocomposite systems reported in the literature. Low strength values are usually related to high porosity and low bond strength. Much of the data to assess the quality of the different composite systems is lacking in the table. For example, the average strength and modulus values of the filaments are required for calculating the theoretical strengths of the composites. Some investigators provide the values given by the manufacturer which could be misleading. A recommended practice is to measure the strengths of several filaments from each lot to obtain an average. One reference (ref 9) provided boron filament T.S. which ranged from 162 kg/mm^2 (231,000 psi) to 300 kg/mm^2 (427,000 psi) for a group of composites.

SUMMARY

Electrodeposition has been shown to be a viable process for producing sound and high strength composites. However, there is room for much improvement in the reproducibility of composites with further advancements in plating techniques and filament making.

From the foregoing, we have learned that successful electrocomposites were produced with the following techniques:

1. Conductive filaments with a diameter of 25 µM (½ mil) or less were simultaneously wound on a mandrel during plating.

2. Nonconductive filaments of 100 µM (4 mils) diameter were unidirectionally wound in single rows with a minimum spacing of one-half filament diameter followed by plating for complete encapsulation. Then, a second row was wound and plated, etc., without removing the mandrel or interrupting electrolysis when the deposit was relatively smooth.

3. Conductive filaments of 100 µM (4 mils) diameter were processed the same as above with two exceptions: minimum spacing was one-filament diameter and the mandrel was removed after each row was plated to remove surface irregularities by machining.

4. Single layers of filaments were also encapsulated by an electrodeposit to form warp sheets or foils followed by hot pressing. In some cases, single filaments were plated with a thin metal and grouped together by hot pressing.

Two of the changes that will significantly improve the quality of electrocomposites are:

1. The use of finer diameter (< 50 μM) monofilaments with high strength and high modulus-to-density ratios.

2. The use of nonconductive filaments when their diameter is greater than 50 μM (2 mils).

Studies are also needed in:

1. Determining the benefits of other pretreatments to improve the wetting and bond strength of filaments.

2. Furthering studies with the use of ultrasonics, periodic reverse, pulse plating, and other aids to determine their influence on voids and properties of the matrix metal.

c. Increase studies on the plating of matrix alloys.

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